Mechanical and Thermal Performance of Cryptostegia Grandiflora Fiber Reinforced Biopolymer Composite Using 3D Printing Technology

A SYNOPSIS

Submitted by

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1. INTRODUCTION

The last three decades have been the golden era of material development in engineering applications, with the use of synthetic fibers increasing in composite products; it provides good strength and a significant contribution to ancillary products. Now that we know it causes high carbon emissions, we are focusing on developing eco-friendly composite products for engineering applications. As a result, natural fiber has the potential to replace synthetic fiber in composite industries while also being biodegradable. Due to its advantageous qualities, such as strength, durability, and elasticity, synthetic fiber is reinforced with a matrix in polymer matrix composites. However, synthetic fiber is non-biodegradable and releases more carbon into the environment. As a result, much research is being conducted on environmentally friendly natural fibers as suitable substitutes for man-made fibers in polymer matrix composites [1].

The natural fibers are composed of cellulose, lignin, hemicellulose, pectin and wax. Natural fibers mainly consist of cellulose, which provides structural stability, stiffness and strength. The crystallinity of natural fibers is based on their cellulose molecules. In addition, the interfacial bonding between the matrix and the fibers makes a major contribution to the mechanical properties of the composites.

The presence of hydroxyl groups (OH) in natural fibers reduces the effectiveness of load transfer between the matrix and the fibers and obstructs proper bonding with the hydrophobic polymer. Besides, the high moisture absorption, low reactivity, hydrophilic nature and compatibility with a polymeric matrix are characteristics of lignocellulose fibers. These characteristics can be improved by applying chemical treatments, such as acetylation, alkalization and silane.

The treatments clean surfaces of natural cellulose fiber by removing impurities and non-cellulosic materials and activating the hydroxyl groups in their surfaces. Both silane and alkali treatments have been reported to be the most efficient and extensively utilized treatments for improving the properties of fibers. Both surface treatments modify the microstructure of the natural fiber and increase the interfacial compatibility among the fiber and matrix [2].

The alkali treatment is the most affordable method for reducing amorphous materials, including hemicellulose and lignin. It also improves the chemical composition, surface morphology, interfacial bonding, thermal stability, crystallinity index and mechanical properties of fibers. It has been reported that treatment of natural fibers with 5% of NaOH improved fiber property changes. The silane treatment is the most efficient method for reducing the volume of hydroxyl with the help of silane coupling agents. The presence of moisture in the fiber helps to form the silanols from the hydrolysable alkoxy group, and these react with the hydroxyl group to produce stable covalent bonds on the fiber surface. The hydrocarbon chains created by the silanols limit the swelling of the fiber [3].

Natural fiber-reinforced polymer composites have gained significant attention in recent years due to their eco-friendly nature and potential for use in various applications. These materials offer a sustainable alternative to traditional synthetic fibers and composites, and they exhibit favorable mechanical properties, low cost, and biodegradability. Natural fibers have been used for centuries in various applications, including textiles, ropes, and construction materials.

The use of natural fiber reinforced polylactic acid (PLA) composites in 3D printing has gained significant attention due to their potential for sustainable and cost-effective applications. These composites offer a promising alternative to traditional materials, with the added benefit of being environmentally friendly. By integrating natural fibers into PLA, researchers have been able to enhance the mechanical properties of the resulting composites, making them suitable for a wide range of 3D printing applications.

In addition to their sustainability and cost-effectiveness, natural fiber reinforced PLA composites also present challenges that require rectifying methods. Researchers have been exploring various techniques to address issues such as fiber-matrix adhesion, dispersion of fibers, and overall composite performance. By understanding and implementing these rectifying methods, the potential of natural fiber reinforced PLA composites in 3D printing can be fully realized, opening up new possibilities for sustainable manufacturing processes [4].

2. LITERATURE REVIEW

Anish Khan et al. [5] extracted and investigated the natural fiber of Cortaderia selloana grass, finding it to have a cellulose content of 53.7%, a crystallinity index of 22%, and a thermal stability of 320 ºC. This study suggests that natural fibers can be used as a promising reinforcement in environmentally friendly composite materials. Vijay et al. [6] conducted a study on Vachellia farnesiana fibers extracted through a 20-day retting process. They evaluated various physical and chemical characteristics, including crystallinity index, thermal stability, and tensile strength. Analytical techniques such as TGA, XRD, and FTIR were used. The study found that the fibers had suitable properties for use as a reinforcing material in lightweight composites. Mabrouk Maache et al. [7] focused on analyzing fibers extracted from the plant Juncus effusus L (JE). The researchers examined various properties of these fibers, such as their morphology, chemical composition, thermal stability, crystallinity, and mechanical properties. The fibers were found to have thermal stability up to 220 °C, making them suitable for use as reinforcement in composite materials. X-ray diffraction revealed that the fibers were semi-crystalline in nature. The fibers also exhibited good tensile strength, elongation at break, and elastic modulus.

Balaji and Nagarajan [8] examined the effects of alkali treatment on cellulose fibers from Sahara aloe vera cactus leaves. Results showed increased cellulose content, decreased hemicellulose, moisture, and wax content, reduced hydrophilic properties, improved compatibility with composite matrices, and improved mechanical properties. The fibers also showed increased thermal stability and decomposition temperature, suggesting their potential as effective reinforcements in polymer composites.

Mohammad Asim et al. [9] studied the effects of different chemical treatments on pine apple leave fibers (PALF) and kenaf fibers (KF) and investigated their influence on mechanical, morphological and structural properties. The fibers were immersed in chemical solutions such as 2% silane solution, 6% NaOH solution and 2% silane and 6% NaOH for 3 hours. The fibers were immersed in various chemical solutions, resulting in improved tensile properties and a reduction in impurities. The silane treatment had the most significant positive effect on tensile properties, morphological changes, and interfacial surface strength (IFSS). The results suggest that silane treatment significantly enhances the properties of PALF and KF.

In their study, Atiqah et al. [10] compared the physical and mechanical properties of sugar palm fibers treated with alkali, silane, or a combination of both. The results showed that the treatments increased tensile strength by breaking down lignin and hemicellulose, while also removing impurities, wax, and pectin. However, the 2% silane treatment roughened the fibers' surface, potentially impacting their interfacial properties and bonding ability. The study suggests that the combined treatments may not be as effective as individual treatments.

Pereira et al. [11] developed a 3D printing filament using rice husks, a waste product from agriculture. They experimented with different weight percentages, finding that adding rice husk fibers increased filament roughness and diameter variation. The study found a decrease in filament density when rice husk content exceeded 15%. The filaments remained stable for fused deposition modeling at temperatures up to 250 ºC. Sarawut Rimdusit et al. [12] examined the impact of polypropylene composites with Hevea Brasilliensis wood flour on thermal and mechanical properties. Results showed that particle size and wood flour content significantly influenced mechanical properties. The study suggests that the optimal size of the particle range for improving properties was 200–300μm.

Durowaye et al. [13] conducted a study to evaluate the mechanical properties of polypropylene composites reinforced with sisal particles to assess their suitability for engineering applications. The composites showed an improvement in hardness, especially with 300 μm sisal particles, reaching a peak value of 3.83 BHN at 25%. However, the toughness decreased as sisal particle concentration increased. The microstructural analysis revealed a well-dispersed sisal particle arrangement, increasing strength and tensile strength. The 150μm particle size was more favorable, making it suitable for interior applications.

Rozman et al. [14] study on oil palm Empty Fruit Bunches (EFB) found an inverse relationship between particle size and flexural strength and modulus. Smaller particles withstand higher loads, with EFB composites with 75-180 μm filler size showing the lowest reduction in flexural strength. Smaller particle sizes also contributed to higher toughness.

Christian Gauss et al. [15] studied 3D-printed polylactic acid composites reinforced with regenerated cellulose fibers. The addition increased filament elasticity,

but formulations with 20% and 30% fiber content showed porosity. The 10% fiber content formulation had the most favorable tensile properties, elongation at break, and thermo mechanical stability, making the composites easy to print.

Wangwang Yu et al. [16] investigated the properties of a PLA/Coffee Grounds Composite designed specifically for 3D printing. In the study, pure PLA powder was mixed with dried coffee grounds in concentrations of 1 %, 3 %, 5 % and 7 %. Different concentrations of coffee grounds were mixed with pure PLA powder. The addition of 3% coffee grounds increased the tensile strength, but no significant difference was found below 3% addition. However, a significant decrease in tensile strength was observed when the addition of coffee grounds increased from 5% to 7%.

The thermal properties of the PLA coffee grounds material were similar to pure PLA. The study also highlighted the economic and environmental benefits of using coffee grounds in PLA filament.

3. RESEARCH GAP

- To improve the production of high-quality, cost-effective and efficient products and remain competitive in the dynamic marketplace, the industry needs to shift from traditional manufacturing methods to advanced manufacturing techniques.
- Among these advanced manufacturing approaches, additive manufacturing (AM) stands out as the most established and important method. Fused Deposition Modeling (FDM) is one of the most commonly used techniques in the field of AM. These advanced manufacturing methods allow designers and engineers to effortlessly create intricate shapes and functional components with improved performance while reducing production costs.
- AM technology offers the flexibility to use a variety of commercially available materials, including high impact polystyrene (HIPS), thermoplastic elastomer (PCTPE), polylactic acid (PLA), transparent polycarbonate, acrylonitrile butadiene styrene (ABS) and nylon.
- This versatility in material options allows for customization and innovation in product design. According to forecasts, the market for filaments, which are crucial for AM, will reach a volume of 6.6 billion dollars by 2026.

- In the industrial context, PLA is proving to be an important polymer alternative to replace petrochemical-based polymers due to its favorable physical and mechanical properties as well as its degradability.
- Currently, PLA serves as one of the main raw materials for desktop 3D printers. However, PLA also has some disadvantages, in particular low impact strength and high brittleness. To overcome these drawbacks, the reinforcement of PLA with fibers or fillers has gained importance, resulting in improved physical, mechanical, degradable and thermal properties.
- The use of natural fiber-reinforced composite filaments in product development has steadily increased in various areas.
- It is important to recognize that ongoing research is essential for the development of new composite filaments with improved properties that match the evolving needs of the industry.
- At the same time, the demand for 3D printing is increasing. There is a need for research into the use of natural fiber-reinforced PLA filaments for 3D printing applications.

4. OBJECTIVE

The objectives of this study can be summarized as follows:

- Develop a procedure for extraction of new natural fiber from the Cryptostegia Grandiflora plant.
- To investigate the Physical, Mechanical, and Thermal stability as well as the Morphology of Raw fibers and fibers treated with 5% alkali and 2% silane solution.
- Develop a procedure for converting the Cryptostegia Grandiflora fiber into filler.
- To fabricate the 5 and 10 wt. % Cryptostegia Grandiflora filler-reinforced Polylactic acid composite filaments.
- Fabricate the different mechanical and thermal study samples using Fused Deposition Modeling 3D printing.
- To study the Physical, Mechanical and Thermal properties of the fabricated samples such as density, tensile strength, flexural strength, impact strength, hardness and water absorption.

5. METHODOLOGY

The research methodology for CGF/PLA composite was shown in Figure 5.1.

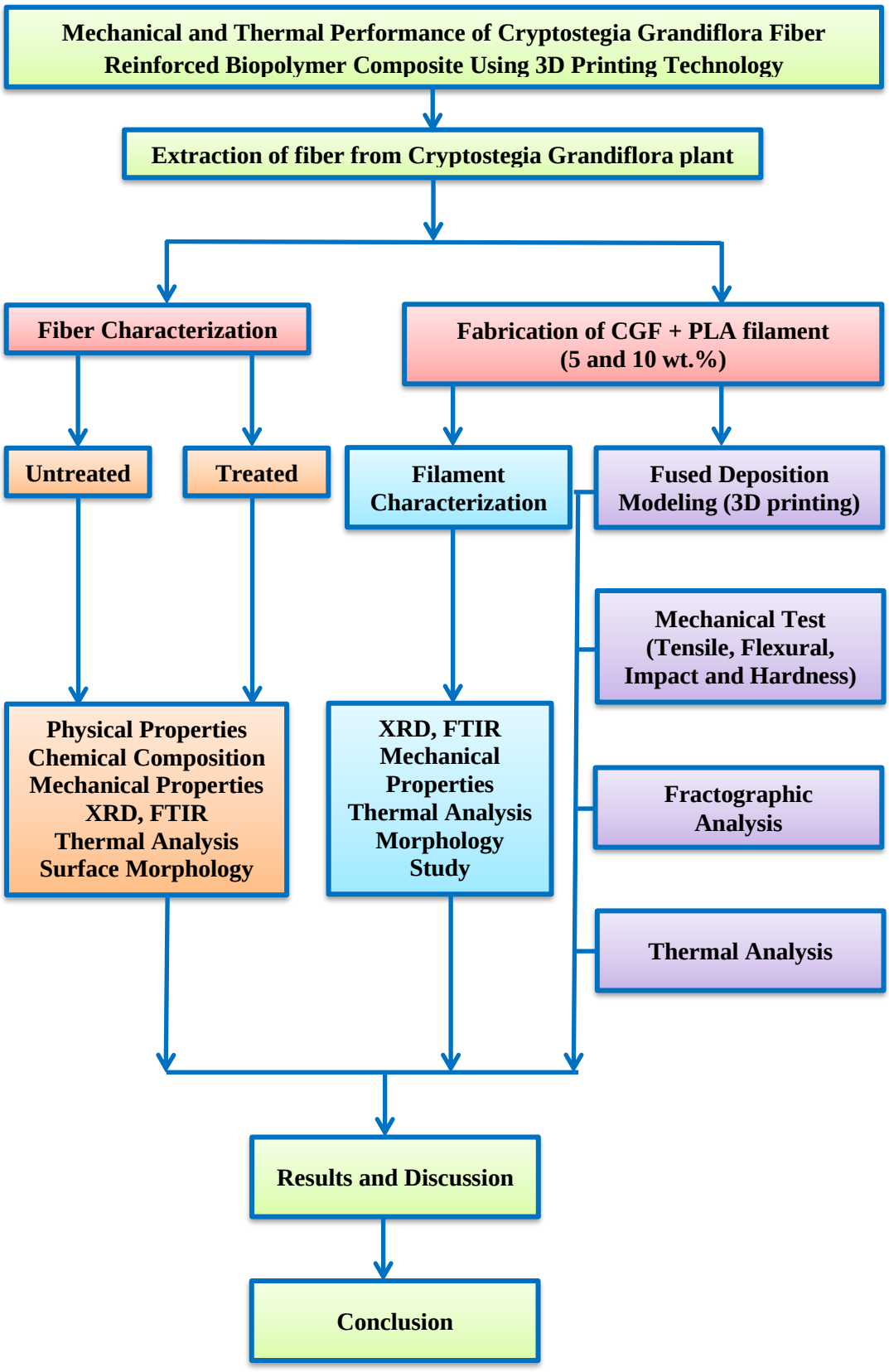

Figure 5.1 Research methodology for CGF/PLA composite

6. MATERIALS AND METHODS

6.1 Materials

Cryptostegia grandiflora (CG), commonly known as rubber vine, is a woody perennial vine native to the southwest Madagascar. It is belongs to Apocynaceae family. While the rubber vine can grow as a shrub to a height of 2 meters (6 feet 7 inches), its climbing nature, supported by surrounding vegetation, enables it to reach impressive lengths of up to 30 meters or 100 feet. It can produce over 8000 seeds in a single reproductive cycle and produce seeds twice every year. It is widespread in worldwide but it is eradicated as it is an invasive species. In particular, the Cryptostegia Grandiflora plant used in this research work was identified in Virudhunagar district, Tamil Nadu, India. Poly lactic acid pellets with a molecular weight of 6.7×10^4 and a density of 1.2 g/cm^3 was used as a matrix. The Cryptostegia grandiflora plant was shown in Figure 6.1.1.

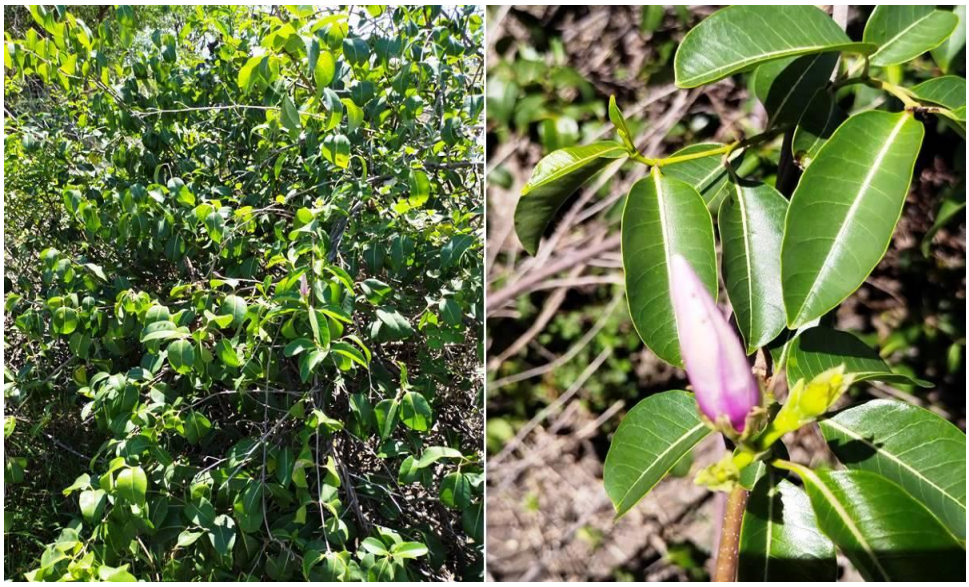

Figure 6.1.1 Cryptostegia Grandiflora plant

6.2 Methods

Fiber Treatment

The dried CGF were immersed in a 5% of NaOH solution for four hours at a room temperature. To prepare the fibers for further analysis, they were cleaned in running water to eliminate any remaining NaOH and dried for three days. To prepare a silane solution, 2% of 3-aminopropyltrimethoxysilane was added to a mixture of

ethanol and water in a ratio of 80:20. Acetic acid was used to adjust the pH value of the silane solution and maintained between 4 and 5. The fibers were then washed and dried for three days at a room temperature after being immersed in the silane solution for four hours.

Powder Making Process

The Cryptostegia grandiflora fiber (CGF) was cut into small pieces with an average length of 1 cm. In a beaker, 30 g of cut CG fiber pieces were immersed in a 10% KOH solution for about 4 hours. The CGF had been kept in a water bath at 90 °C for an hour. The treated CGF was then allowed to cool in the ambient temperature. The powder was produced by grinding the processed fiber after it had been washed and heated in a hot air oven temperature at 60°C for duration of two hours. The grinded CGF powder was used as a filler in filament production.

Filament Fabrication process

The fabrication process of the polylactic acid composite filament is shown in Figure 6.2.1.

Figure 6.2.1 Filament fabrication process

To initiate the process, PLA pellets and CG filler are manually blended. Subsequently, a twin-screw extruder, specifically the one from Brabender in Duisburg, Germany, featuring a 32 mm screw diameter and a 16:1 L/D ratio, is employed for the

initial mixing of the composite materials. The optimal temperature to achieve smooth extrusion and optimum filament diameter was determined to be between 170 to 200 °C. The haul-off speed is meticulously adjusted to control the diameter of the filament strand. This comprehensive procedure results in the production of 5 and 10 wt. % CGF + PLA filaments with a consistent diameter of 1.75 ± 0.05 mm. This approach ensures a systematic and controlled production of composite filaments, meeting the desired composition and diameter specifications for subsequent applications.

7. RESULTS AND DISCUSSION

7.1 Raw Fiber Characterization

The density of raw CGF is 1.02 g/cc, which is important for developing lightweight composites. The fiber has a high cellulose content of 79.20%, significant hemicellulose content of 17.20%, and low wax content of 0.54%, contributing to its strength, stiffness, and fire-resistance. The calculated crystallinity index of CGF is 62%, indicating its potential as a reinforcement material for composites. FTIR analysis confirms the presence of cellulose, wax, hemicellulose, and lignin in CGF. The TGA curve of Cryptostegia Grandiflora fiber reveals its thermal stability up to 230 ºC, as well as the gradual mass loss up to 340 ºC and the decomposition of hemicelluloses, pectin, and cellulose glycoside bonds. Figure 7.1.1 illustrates the tensile properties of the CG fiber.

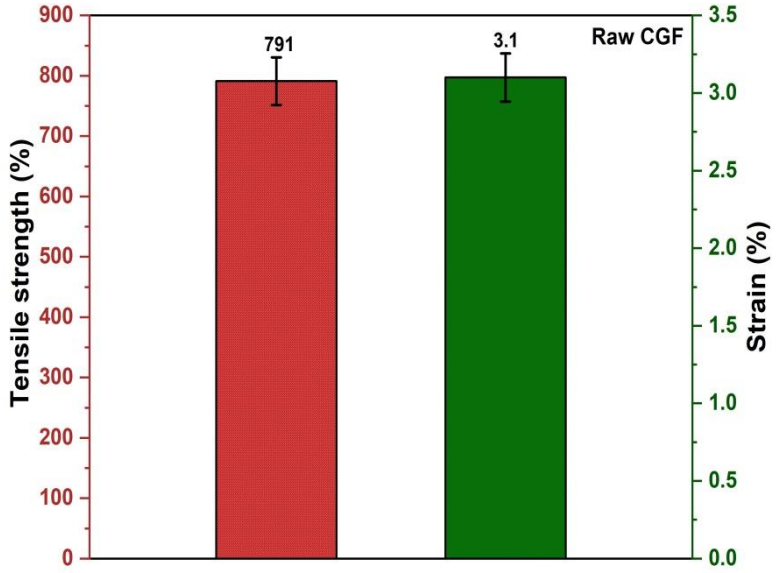

Figure 7.1.1 Tensile strength of Raw CGF

7.2 Treated Fiber Characterization

Chemical treatments, specifically alkali and silane treatments have been used to improve the alignment and packing of cellulose fibers. The tensile strength of the treated and untreated CGF is shown in Figure 7.2.1.

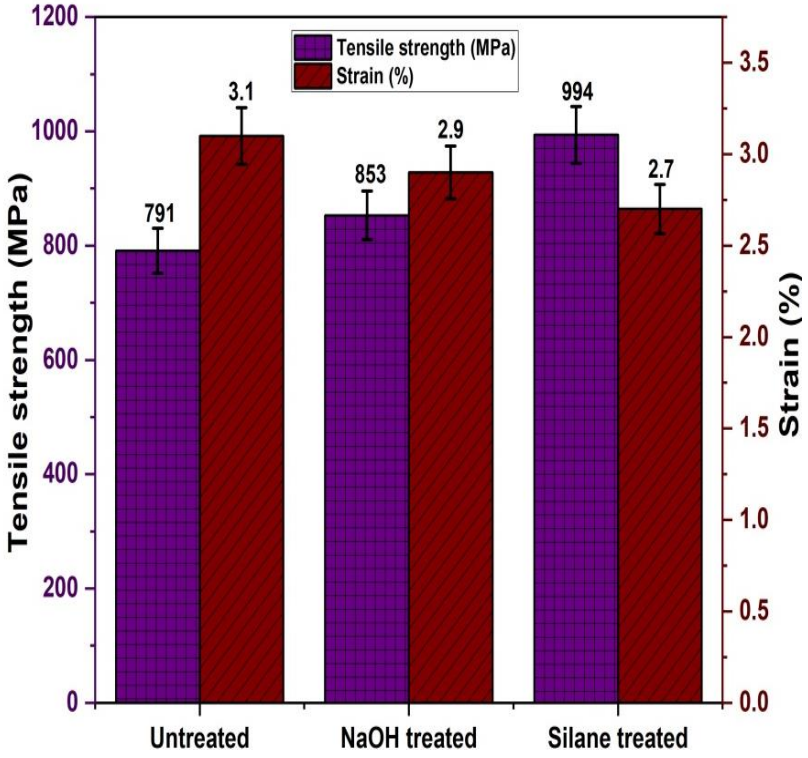

Figure 7.2.1 Tensile strength of treated and untreated CGF

This leads to increased crystallinity and density in cellulose-based materials. The densities of silane and NaOH treated cellulose-based materials increase by 27% and 44% respectively, compared to untreated materials. This is achieved by removing hemicelluloses and lignin and densifying the cell walls. Silane treatment increases the cellulose content by 10% and reduces the hydrophilic behavior, hemicellulose, and ash content of natural fibers. The treated fibers also exhibit improved tensile strength, with a 25% increase for silane-treated fibers and a 7% increase for NaOH-treated fibers, compared to untreated fibers. The thermal stability of the treated fibers is also improved, as shown by lower weight loss in TGA analysis and improved degradation behavior. The surface morphologies of treated and raw CGF were compared using SEM, revealing that chemical treatments such as alkali and silane result in roughened, cleaned, or smoothed fiber surfaces. Figure 7.2.2 depicts the TGA curve for the treated and untreated CGF.

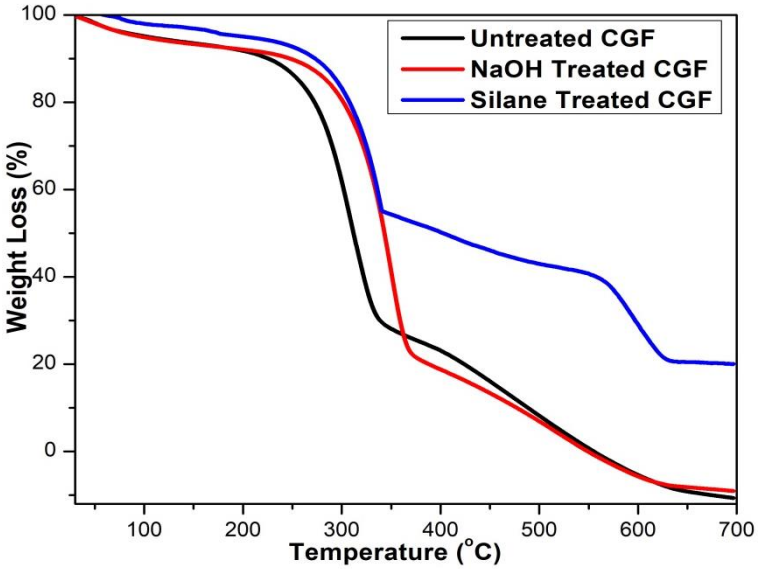

Figure 7.2.2 TGA curve of untreated and treated CGF

7.3 Filament Characterization

The table 7.3.1 summarize the mean values of the mechanical characteristics of PLA and composite.

Table 7.3.1 Tensile properties of PLA, 5 and 10 wt. % CGF + PLA Filaments

Filament	Diameter (mm)	Tensile strength (MPa)	Strain (%)	Young's Modulus (GPa)
PLA	1.75 ± 0.05	43.62	4.04	2.3
5 wt.% CGF + PLA	1.75 ± 0.05	54.00	3.68	3.77
10 wt.% CGF + PLA	1.75 ± 0.05	49.05	3.2	2.7

The addition of Cryptostegia Grandiflora (CG) filler to PLA filaments has several effects on the composite material. Firstly, the addition of 5 and 10 wt.% of CG filler decreases the melt flow index (MFI) of the composite, resulting in a decrease of 8 and 14% in MFI. This decrease in MFI indicates an increase in melt viscosity, which is beneficial for FDM printing and allows for the printing of parts with overhang features using low melt viscosity filament. Additionally, the addition of 10 wt.% CG filler to PLA composite filament improves the heat resistance of the material. It increases the initial decomposition temperature and leads to a different pattern of weight loss and residue formation compared to pure PLA, as observed through thermal

analysis. Morphological investigation of the CG filler reveals that the majority of particles are less than 100 µm in size. The 5 and 10 wt.% composite filaments exhibit slightly rougher and less uniform surfaces compared to pure PLA filaments. There is also visible formation of an effective interface between the matrix and the CG fillers, but slight disruptions such as crack formations around CG filler clusters. Furthermore, the inclusion of CG filler significantly enhances the tensile strength of composite filaments.

7.4 Physical, Mechanical and Thermal Properties of 3D Printed Composite

The mechanical properties of 5 and 10 wt.% CGF/PLA composite and pure PLA are shown in Figure 7.4.1.

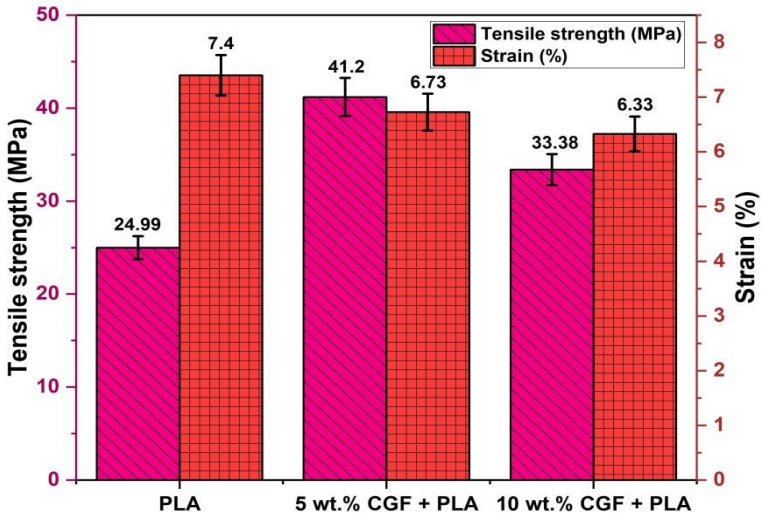

Figure 7.4.1 Tensile strengths of PLA, 5 and 10 wt.% CGF/PLA filament composite

The addition of CG filler as a reinforcement to polylactic acid (PLA) resulted in decrease the density by 1% and 5.2% for 5 and 10 wt.% CGF/PLA composites, due to the closed morphology and lower density of the CGF. The addition of CG filler to PLA resulted in improved tensile strength but decreased elongation at break due to good interfacial adhesion and stress transfer. With the 5 wt.% composite showing the highest tensile strength, and the 10 wt.% composite exhibiting lower tensile strength and elongation due to challenges in achieving uniform dispersion. The incorporation of cryptostegia grandiflora filler into PLA composites enhances flexural strength, impact resistance, and hardness. The improved properties are attributed to enhanced interfacial adhesion, dispersion of micron-sized CG particles within the matrix, and interfacial bonding facilitated by fiber surface treatment. The use of micron-sized fillers in PLA

enhances the mechanical properties by promoting extensive hydrogen bonding with the matrix, resulting in improved adhesion and stress transfer between the fillers and the matrix, as evidenced by the fractured surfaces and SEM analysis. The inclusion of 5 and 10 wt.% CG filler in PLA filament leads to an increase in water absorption compared to pure PLA, indicating the hydrophilic nature of CG filler, as confirmed by FTIR study, and suggesting decreased water resistance of composite. The morphology of tensile fractured surfaces of both PLA and CGF/PLA composite samples are shown in Figure 7.4.2.

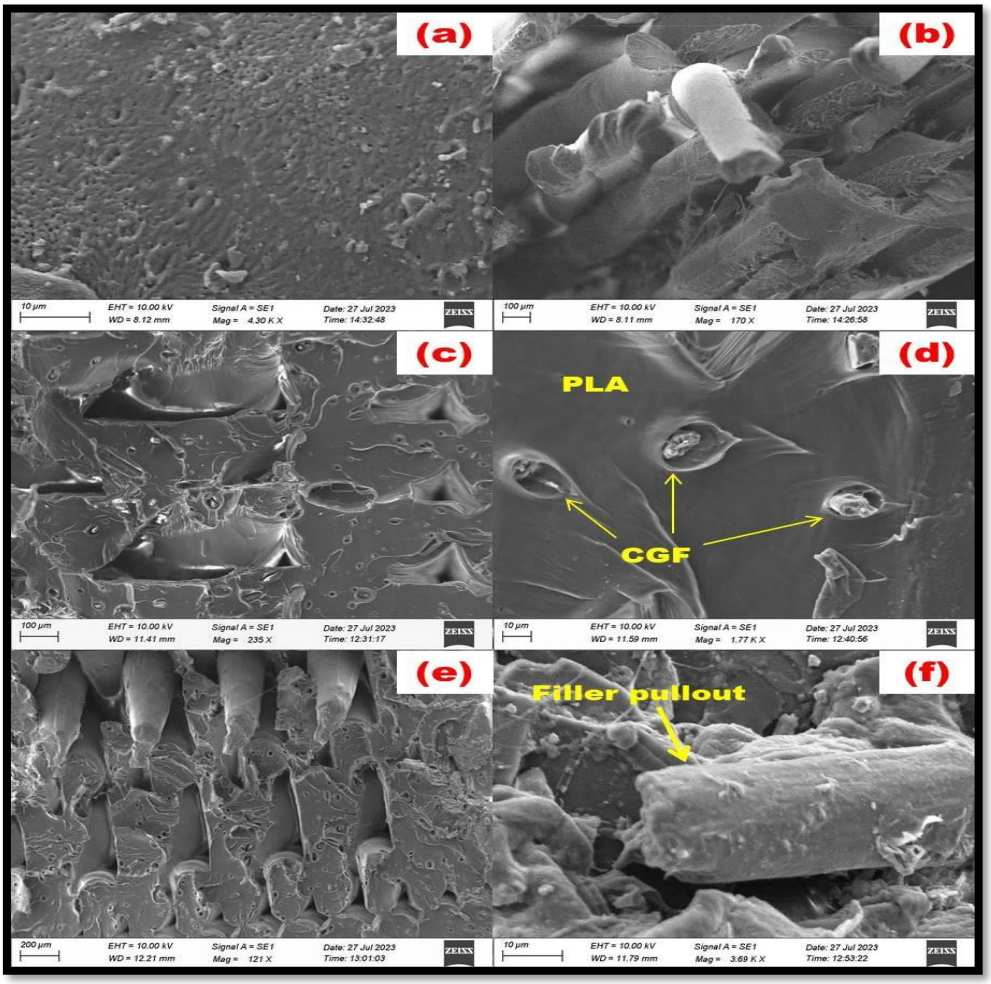

Figure 7.4.2 Tensile fractured SEM images (a - b) Pure PLA (c - d) 5 wt.% CGF/PLA (e - f) 10 wt.% CGF/PLA

The experimental results of the cryptostegia grandiflora filler reinforced composite samples strongly recommend their potential application in the aviation, sports, and automotive industries due to their mechanical, thermal, and absorption properties, as well as surface morphologies.

8. ORGANIZATION OF THESIS

Chapter 1: In this chapter, natural fiber-reinforced polymer composites are introduced, with a focus on their mechanical and thermal properties as well as insights into characterization studies. Furthermore, the chapter delves into an overview of 3D printing, exploring its various types, the customary materials used in 3D printing, and the application of natural filler-reinforced composites within this technological framework.

Chapter 2: This section references relevant literature to explore the characterization of plant fibers, composite materials designed for 3D printing, the use of natural fillers for reinforcing 3D printing filaments, and the subsequent performance analysis. A comprehensive review of pertinent literature serves as the foundation for identifying research gaps and requirements. Furthermore, the section outlines the research objectives, providing clarity on the specific aims that steer the study.

Chapter 3: This section describes the materials and experimental procedures used in this study. This chapter gives important details on the processes used to characterize and extract natural fiber using techniques including FTIR, XRD, TGA, and morphology. Also, it includes test specifications, test methods, types of equipment, and standards that are used to test filament and FDM 3D-printed samples.

Chapter 4: This chapter offers detailed characterization studies of the newly extracted Cryptostegia grandiflora fiber under raw and treated conditions for light-weight composite materials.

Chapter 5: This section describes the composite filament fabrication method, the characterization of fabricated filaments, and mechanical and thermal analysis.

Chapter 6: This chapter offers a detailed analysis of the experimental findings of the FDM 3D printed samples.

Chapter 7: This chapter defines the conclusion based on discussions such as fiber and filament characterization, the results of empirical studies of FDM 3D printed samples, and the future scope of research.

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LIST OF PUBLICATIONS

1. **A.Udhayakumar**, K. Mayandi, N. Rajini, R. K. Devi, M. Muthukannan, and M. Murali, Extraction and Characterization of Novel Natural Fiber from Cryptostegia Grandiflora as a Potential Reinforcement in Biocomposites, *Journal of Natural Fibers*, **20** (1) (2022), 2159607.

2. **A.Udhayakumar**, K. Mayandi, N. Rajini, R. Kanniga Devi, M. Muthukannan, M. Murali, Sikiru O Ismail, Faruq Mohammad and Hamad A Al-Lohedan, Effect of chemical treatment on physico-chemical properties of a novel extracted cellulosic Cryptostegia grandiflora fiber, *Materials Research Express*, **10** (7) (2023), 075508.

3. **Udhayakumar Arumaiselvan**, Mayandi Kalimuthu, Rajini Nagarajan, Murali Mohan, Sikiru O. Ismail, Faruq Mohammad, Hamad A. Al-Lohedan, and Kumar Krishnan. Mechanical, Physical and Thermal Properties of Polylactic Acid Filament Composite Reinforced with Newly Isolated Cryptostegia grandiflora Fiber. *Bio Resources* **19** (2) (2024) 3740-3754.

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